

Non-destructive and micro-invasive techniques for cultural heritage diagnostics

- a case study of glazed tiles from Portuguese historical buildings

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Among cultural assets, ceramics and particularly glazed tiles ("azulejos" in Portuguese and Spanish, from the Arab designation "al-zuléija" or "al-zulaiju") deserve particular attention in the Mediterranean region, where they have long been used to decorate buildings. "Azulejos" are present in many historical Portuguese buildings of the XVII to the XIX centuries. In fact, one may say that "azulejos" are everywhere in Portugal. They decorate everything from walls of churches and monasteries, to palaces and ordinary houses. Most of these "azulejos" present various degradation features, mainly due to exterior exposure in a range of different environments.

Scientific analysis / investigation of cultural heritage objects is ideally conducted in a manner that enables fail-safe transfer to late generations. A research methodology including the application of non-invasive (neutron tomography –NT) and micro-invasive techniques (XRF, XRD) for diagnostic of two types of "azulejos" is presented.

Materials studied: - Glazed tiles from the XVII century

Madre de Deus Church, Lisboa (sample MD)



Nossa Senhora dos Aflitos Church, Elvas (sample NSA)

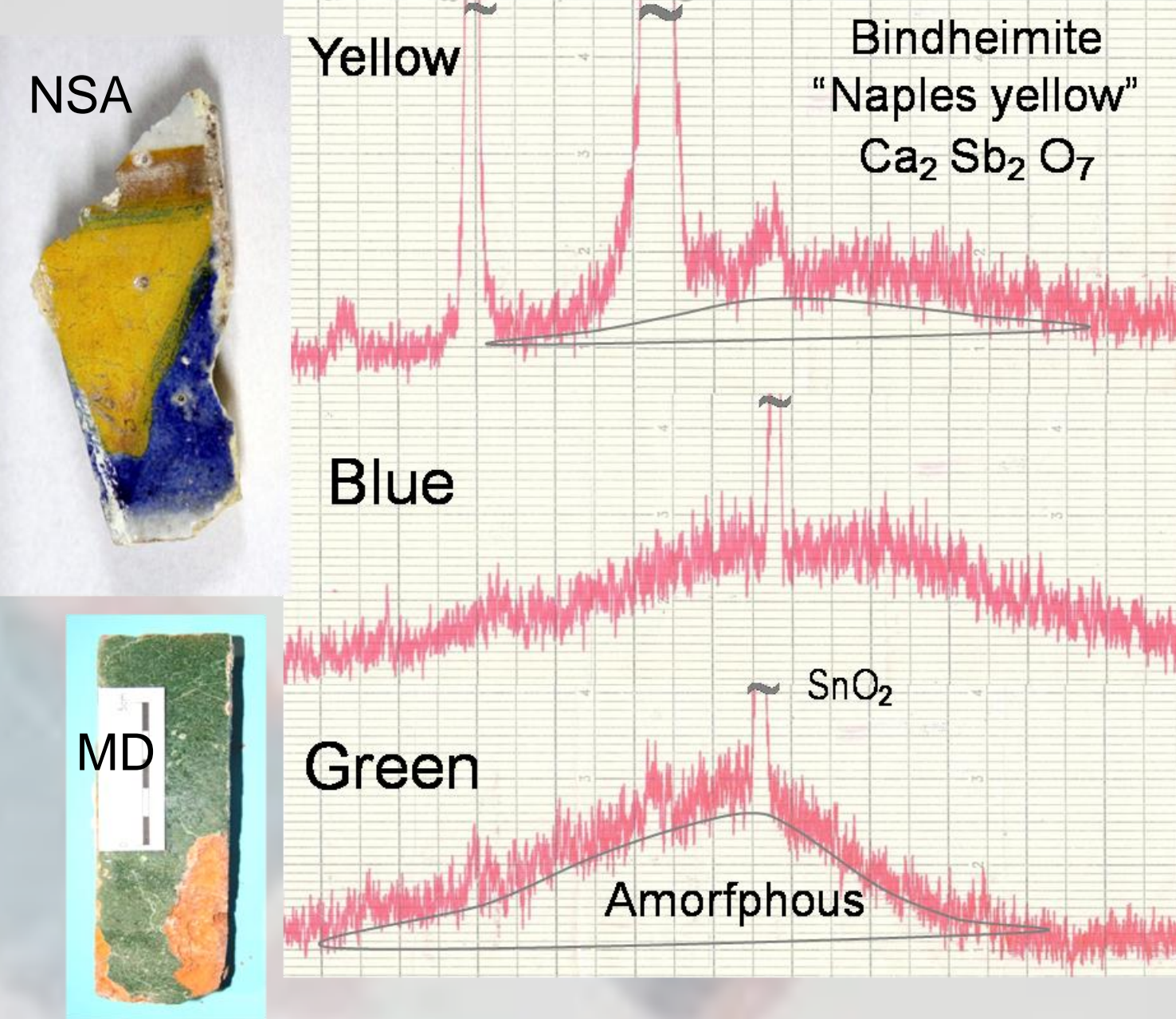


Glaze

Chemical characterization by XRF

Sample	Yellow NSA	Blue NSA	Green MD
K Ka	136.8*	56 020	187 933
K Kb	118.2	11 642	19 971
fundo	134.0	548	1 109
Sb La	117.5	30 943	3 526
Sn Lb	114.4	3 469	2 352
Ca Ka	113.1	26 299	48 335
fundo	71.00	328	569
Cr Ka	69.36	558	678
Fe Ka	57.52	66 112	105 667
Co Ka	52.80	2 451	32 702
fundo	50.00	1 176	1 306
Cu Ka	45.03	2 904	5 375
Zn Ka	41.80	21 079	37 798
As / Pb	34.00	129 738	394 215
As Kb	30.45	2 327	7 034
Pb Lg	24.07	14 670	33 446
fundo	21.50	1 809	1 605

Phases identification by XRD



BIOBURDEN ANALYSES OF TILES

METHODS:

1) DESTRUCTIBLE – MECHANICAL destruction of samples item proportions

Wash of the samples in physiologic serum with Tween 80 (0.1%) and homogenization by Stomacher® (mechanical procedure). Spread aliquots of washing solution in Tryptic Soya Agar dishes and incubation at 30 °C for seven days. Colony forming units (c.f.u.) were counts along this period.

2) Swab – Swab scrub samples portions with a swab in 5 ml of physiologic serum with Tween 80 (0.1%).

Homogenize the swab in the wash solution by vortex (3000 rpm) during 1 minute.

Spread aliquots of washing solution in Tryptic Soya Agar dishes and incubation at 30 °C for seven days. Colony forming units (c.f.u.) counts along incubation period.

METHOD EFFICIENCY

DESTRUCTIVE: 60 – 70%

SWAB: 40 – 70%

BIOBURDEN :

NSA : $(9 \pm 4) \times 10^2$ UFC/cm²

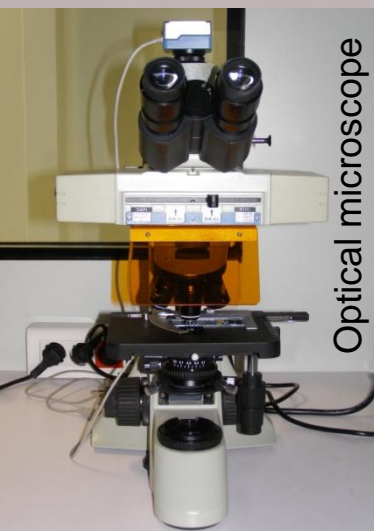
MD : 4.1 ± 0.8 UFC/cm²

BIOBURDEN CHARACTERIZATION (n = 169 isolates)

MOST FREQUENT MORPHOLOGICAL TYPES

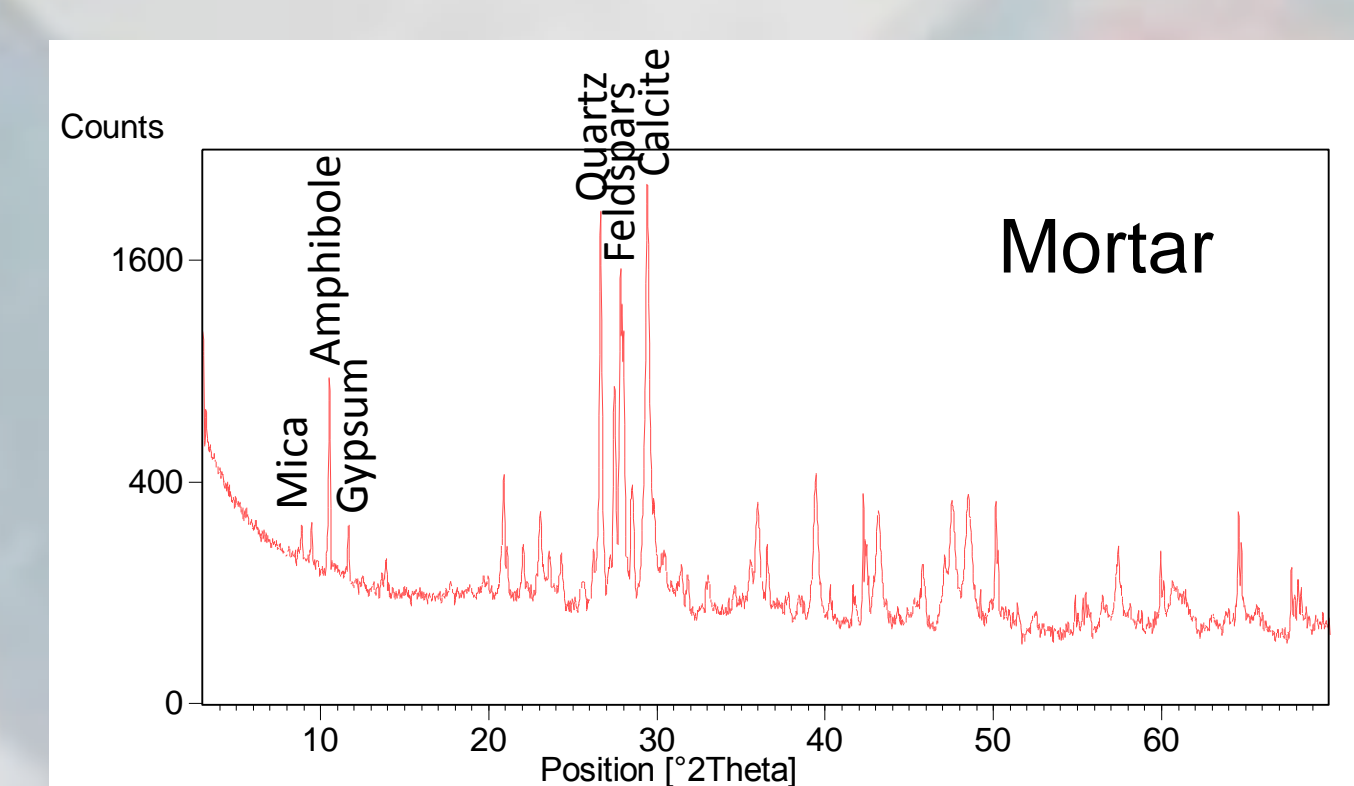
NSA: Gram positive cocci, catalase positive (95%)

MD: Gram negative rods, oxidase positive (64%)

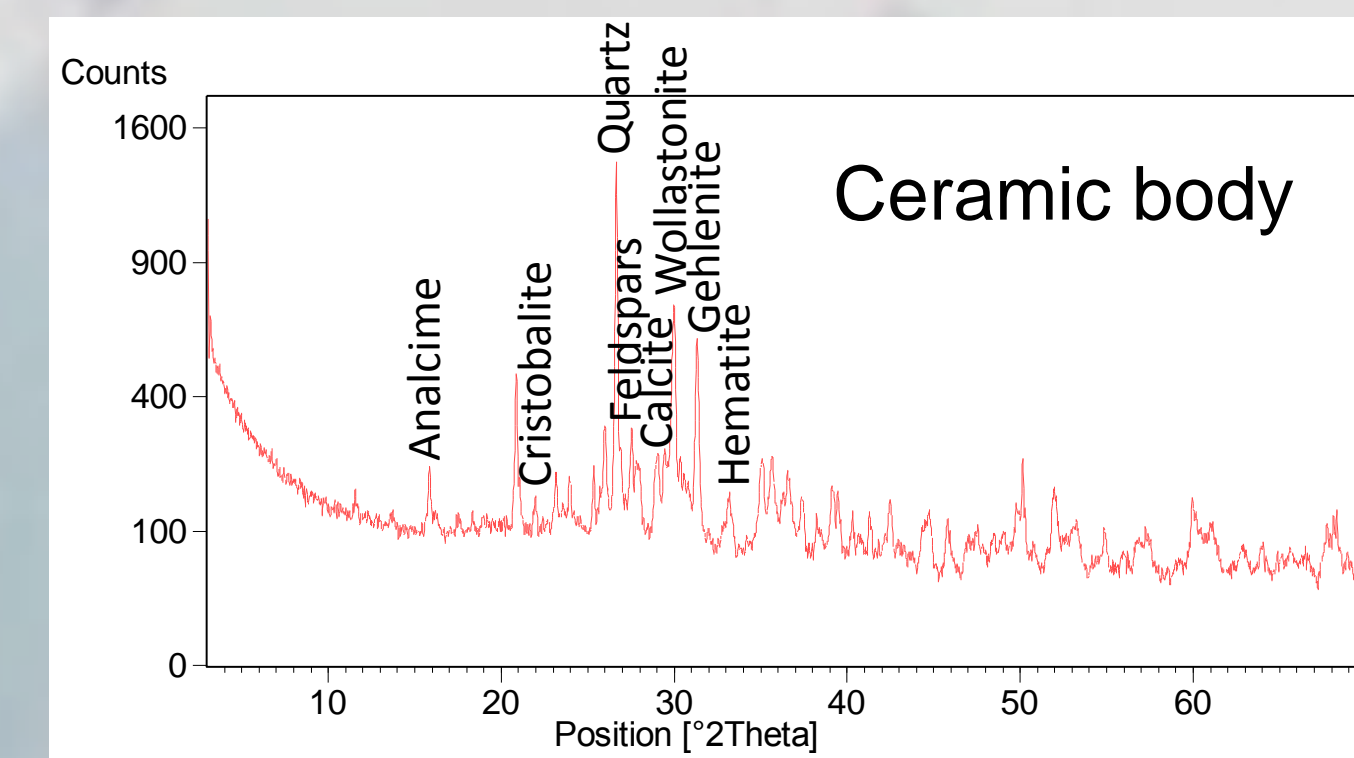


Ceramic body and mortar

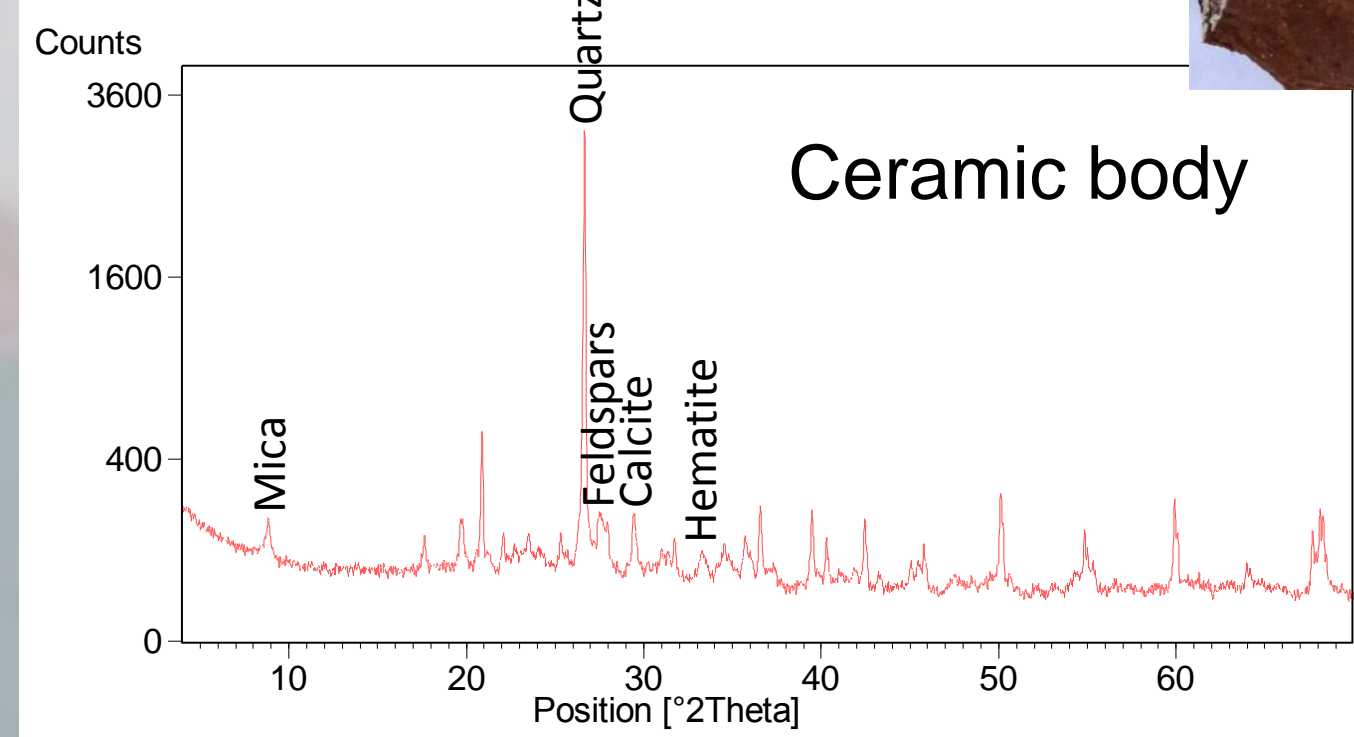
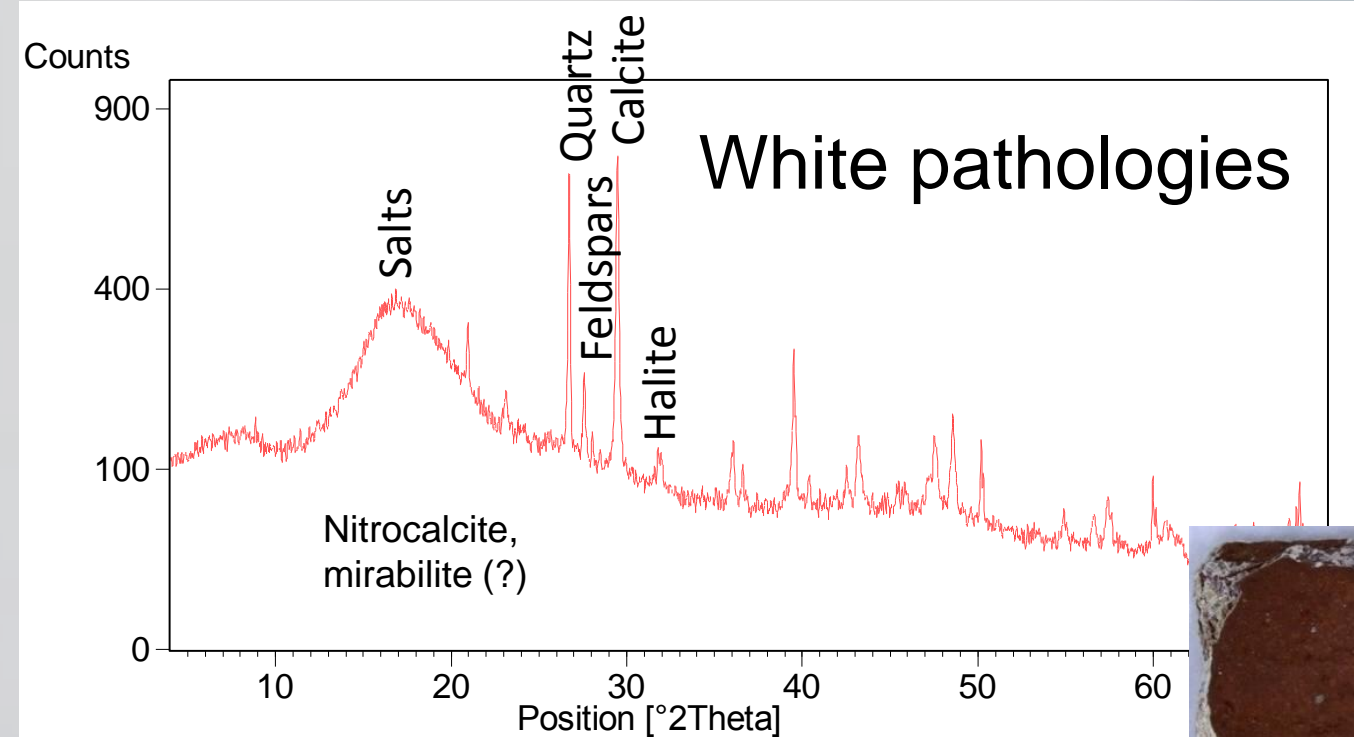
Phases identification by XRD



NSA



MD



Neutron Tomography (NT)

Visualization of physical structures in the interior of an object without physically opening.

Neutrons have the advantage to penetrate most metals (especially heavy ones) very efficiently and to detect hydrogenous compounds very sensitively.

NT has application fields in moisture detection, test adhesive connections, structure analysis

Neutron radiography has a tradition since neutron sources and imaging devices became available. Although this method cannot compete directly with X-ray methods on a broad scale due to a smaller number of strong neutron sources (which are mainly stationary ones, such as research reactors or spallation sources), it has specific advantages:

neutrons have a high interaction probability with hydrogen (and other light elements like carbon) and a lower attenuation in several heavy elements which are “black” for X-rays, such as Pb, Bi and U.

